

PATENT  
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Aqueous Dispersions for the Hydrophobic Finishing of Fibers and  
Flat Textiles

This invention relates to aqueous dispersions containing fluorinated and non-fluorinated poly(meth)acrylates and to the use of such dispersions for the hydrophobic finishing of fibers and flat textiles.

It is known and common practice in the textile industry to treat fibers  
5 and flat textiles with aqueous dispersions containing fluorine-containing copolymers based on perfluoroacrylates. Perfluoroacrylates provide textile materials with both strongly hydrophobic and strongly oleophobic properties. Perfluoroacrylates such as these are described, for example, in  
10 **DE 1 247 659. EP 1 146 103 A1** also describes aqueous dispersions containing polyfluorinated polymers which are used for finishing textiles.

Although the finishing of textiles with perfluoroacrylates or other suitable polyfluorinated compounds does lead to the desired properties of the textile materials, the fluorinated compounds are still very expensive. Accordingly, there is a need to make the hydrophobic finishing of textiles  
15 less expensive without losing the effectiveness of the finish.

It has now been found that, by combining polymeric perfluoroacrylates known per se with non-fluorinated polymeric acrylates, the proportion of the expensive fluorinated products can be reduced without diminishing the effectiveness of the hydrophobic finish.

20 Accordingly, the present invention relates to aqueous dispersions containing at least:

- a) a copolymer of compounds corresponding to formula (I):  $R^1-O-CO-CR^2=CH_2$  (I), in which  $R^1$  is a branched or unbranched alkyl group

containing 8 to 22 carbon atoms and  $R^2$  is a hydrogen atom or an alkyl group containing 1 to 4 carbon atoms, with monomers selected from compounds corresponding to formula (Ia):  $R^3-O-CO-CR^4=CH_2$  (Ia), in which  $R^3$  is a branched or unbranched alkyl group containing 1 to 6 carbon atoms and  $R^4$  is a hydrogen atom or an alkyl group containing 1 to 4 carbon atoms, and

- b) a copolymer of compounds corresponding to formula (II):  $C_nF_{2n}-CH_2-CH_2-O-CO-CR^5=CH_2$  (II), in which  $R^5$  is a hydrogen atom or an alkyl group containing 1 to 4 carbon atoms and  $n$  is a number of 4 to 12, with monomers selected from compounds corresponding to formula (Ia), and
- c) emulsifiers, with the proviso that the ratio by weight between the copolymers a) and the copolymers b) is 10:1 to 1:10.

Both the compounds of formula (I) and (Ia) and the compounds of formula (II) are known per se. They are derivatives, more particularly esters, of acrylic acid or methacrylic acid.

The perfluorinated compounds corresponding to formula (II) are generally prepared from tetrafluoroethane which is polymerized and iodized and converted into a perfluorinated alcohol in a Grignard reaction with ethylene oxide. In a final reaction step, the perfluorinated alcohol is reacted with acrylic acid or methacrylic acid to form the required perfluorinated acrylates or methacrylate esters. When it comes to selecting the compounds corresponding to formula (II), it is of advantage to use perfluorinated compounds of formula (II) where the index  $n$  is a number of 6 to 10.

The compounds of formula (I) are also known esters of acrylic or methacrylic acid with fatty alcohols, the fatty alcohols having to contain 8 to 22 carbon atoms.

The monomers of formulae (I) and (II) independently of one another

are reacted with the acrylates or methacrylates of formula (Ia), generally by emulsion polymerization in aqueous medium, to form copolymers using emulsifiers c), after which the two components a) and b), which already contain the emulsifiers from the production process, are mixed to form the  
5 dispersions according to the invention.

The emulsion polymerization is a special polymerization process in which water-insoluble monomers are emulsified in water with the aid of emulsifiers and polymerized using water-soluble initiators (for example potassium persulfate; redox initiators). Particularly preferred monomers of  
10 formula (Ia) are selected from the group consisting of methyl, ethyl, n-propyl, i-propyl and/or butyl esters of acrylic or methacrylic acid. The compounds of formula (Ia) independently of one another may be used for the preparation of polymer dispersions a) and b). In the polymerization reaction to form components a) and b), it is of advantage for the  
15 percentage of monomers corresponding to formula (I) and (II) to be at least 50% by weight and preferably at least 70% by weight, based on the comonomers (Ia). It is also possible to polymerize only monomers of formulae (I) and (II). However, the percentage of comonomers of formula (Ia) in the polymerization is preferably at least 5% by weight.

20 According to the invention, the ratio by weight between components a) and b) must be between 10:1 and 1:10 and preferably between 5:1 and 1:3. The range from 3:1 to 1:1 is particularly preferred.

The dispersions according to the invention must contain emulsifiers c) for stabilization. The emulsifiers are preferably selected from nonionic  
25 and/or cationic emulsifiers. In a particularly preferred embodiment, the emulsifiers are cationic emulsifiers, i.e. compounds containing a positive charge in the form of a quaternized ammonium group or an amine group with a neutral charge which change into the ionically protonated form on acidification of the system as a whole. Examples of such cationic  
30 compounds can be found in **WO 88/00991**, pages 2 and 3. However, other

quaternized compounds known to the expert, more particularly so-called esterquats, i.e. quaternized alkyl ammonium compounds containing at least one ester group in the alkyl group, are also suitable for stabilizing the dispersions according to the invention.

5           The aqueous dispersions according to the invention contain components a) and b) in total quantities of 0.1 to 50% by weight and preferably in total quantities of 1 to 35% by weight, based on the total weight of the aqueous dispersion. The emulsifiers c) are present in quantities of 0.1 to 10% by weight and preferably in quantities of 0.5 to 5%  
10 by weight, based on the total weight of the dispersion. It can be of advantage to use water-miscible organic solvents besides components a) and b) and the emulsifiers c). Preferred water-miscible organic solvents are polyols containing 2 to 6 carbon atoms and 2 to 6 hydroxyl groups, alkyl ethers or partial alkyl ethers of these polyols or the completely or partly  
15 alkoxyated derivatives of the polyols or acetone.

          The dispersions according to the invention are prepared by first mixing an aqueous cationically or nonionically stabilized dispersion a) with an aqueous cationically or nonionically stabilized dispersion b). The mixture thus obtained may be applied to textiles and fibers by suitable  
20 methods known per se. Suitable textile materials are both cotton and polyester or polyamide fibers and blends. The aqueous dispersions according to the invention are applied to the fibers or to the textile material by conventional techniques, for example using a padding roller. The concentration of active substance is preferably between 1 and 5 g per liter,  
25 based on the liquor.

          The present invention also relates to the use of the aqueous dispersions described in the foregoing for the hydrophobic finishing of fibers and flat textile materials.